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Outgassing rates of PEEK, Kapton® and Vespel® foils

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Summary

The outgassing rates of PEEK, Vespel® and Kapton® foils as a function of time and thickness have been studied through a series of pump-down tests. Samples of at least five different thicknesses for each material, exposed to normal levels of moisture in the air (30-65% R.H.), were pumped in a dedicated setup. The pump down curves were fitted with a Fickian diffusion model. A discrepancy between the model and the experimental results was observed for extended pump down time. A new empirical model was proposed and applied with excellent results. The discrepancy could be due to micro-voids in the vitreous parts of the polymers as described by the dual model.

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1 INTRODUCTION

Polymeric materials have been historically avoided in high vacuum (HV) and ultra-high vacuum (UHV) systems due to their high outgassing rates [1] [2]. However, their use is sometimes unavoidable because of manufacturing, technical or economic reasons: polymers such as PEEK (polyether ether ketone), Vespel® and Kapton® (polyimides) are often found in vacuum equipment as connector pins, gaskets, tapes and insulating layers of electrical cables. Understanding the outgassing behaviour of these materials is therefore important to quantify their impact on the quality of the vacuum in HV and UHV systems.

Unfortunately, only a few sparse studies have been conducted so far on this subject. In 1967, Hait [3] investigated the outgassing behaviour of gaskets made of polyimide and discovered that the material released significant amounts of water vapour after being exposed to air, due to its high hydrophilicity. A study conducted by Chiggiato et. al. [4] showed that also PEEK releases mainly water molecules under vacuum, even after several hours of bakeout at mild temperatures (100-150°C). This behaviour was again observed in PEEK, Vespel® and Kapton® samples during subsequent studies by Battes et. al. [5] and Riihimäki [6]. In another study on the solubility and diffusion of water in PEEK, Grayson [7] described the sorption and desorption processes in this polymer in terms of Case I Fickian diffusion; likewise, Han et. al. [8] found that the sorption and diffusion mechanisms in polyimide films were nearly Fickian. Although polymers might often deviate from this behaviour [9, p. 255], especially when large amounts of permeant cause significant changes in the polymer structure (swelling), it seems at least reasonable to assume that, at low concentrations, the outgassing of the mentioned semi-crystalline polymers could be satisfactorily described by the Fickian diffusion model. The latter has been extensively studied, and useful equations to describe the diffusion limited outgassing process have been formulated for a number of special cases [9, pp. 1-253]. With the present study, the authors aim to characterize the outgassing behaviour of selected semi-crystalline polymers exposed to normal levels of moisture in the air before pump-down. The results of several measurements will be presented and the suitability of the Fickian diffusion model for estimating the outgassing rates of such polymers will be tested and discussed.

2 SELECTED MATERIALS

Due to their relevance for vacuum technologies, PEEK, Vespel® and Kapton® polymers were selected for this study, more specifically the unfilled grades Kapton® HN, Vespel®SP-1, and PEEK 450G, the first two produced by DuPontTM and the latter produced by VictrexTM. All three materials were purchased from the supplier Goodfellow¹.

The PEEK and the Vespel® were shipped as rods with diameters of 51 mm and 50.8 mm, respectively. The Vespel® rod was cut into 5 disks with thicknesses from 0.25 mm to 1.80 mm, while 7 disks with thicknesses from 0.16 mm to 1.28 mm were obtained from the PEEK rod; the Kapton® was instead received as a set of square sheets with side length of 100 mm and thicknesses ranging from 0.0125 mm to 0.125 mm (Figure 1). Since the disks of PEEK and Vespel® had to be machined on a lathe, they were cleaned with anionic surfactant agent before the tests. The Kapton® sample sheets were instead tested as received from the provider. A

¹ https://www.goodfellow.com

summary of the final dimensions and shapes of the samples is provided in Table 1.

Polymer	Manufacturer	Sample	Side / diameter	Faces surface ²	Thicknesses
		shape	(mm)	(cm^2)	(mm)
PEEK	Victrex TM	Disk	51	40.9	0.16, 0.18, 0.47, 0.50,
450G	Victiex 1 M				0.72, 0.94, 1.28
Vespel®	DuPont TM	Disk	50.8	40.5	0.25, 0.45, 0.92, 1.30,
SP-1	Duroni				1.80
Kapton®	DuPont TM	Square sheet 100	200	0.0125, 0.025, 0.05,	
HN	Duroni		100	200	0.075, 0.125

Table 1: Information on the polymer samples

PEEK, Kapton® and Vespel® are all very good electrical insulators; they have high tensile strength and stiffness and display much higher thermal resistance than most of the other thermoplastics. PEEK is a linear aromatic polymer which melts at 343°C and has a useful operating temperature up to 260° [10] C. Kapton® and Vespel®, both polyimides [11] [12], do not exhibit any melting point up to 400°C, temperature above which they thermally degrade [13]. PEEK can be processed via traditional extrusion and injection moulding methods, while the polyimides, due to the lack of a melting point, must be either produced directly as a film (Kapton®) at the synthesis stage [14], or shaped with sintering techniques (Vespel®) such as direct forming, ram extrusion, etc. [12]. The amount of water (in mass percentage) contained in these materials was expected to be 0.31-0.45% for PEEK [15] [10], 1-1.3% for Vespel® [12] and 1.3% for Kapton® [15].

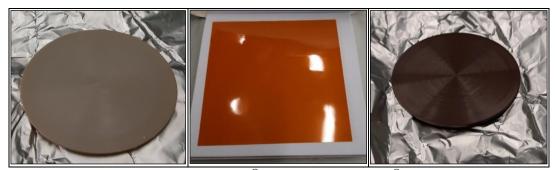


Figure 1: PEEK (left), Kapton® (centre), and Vespel® (right) samples

3 EXPERIMENTAL METHODS

3.1 MEASURING EQUIPMENT

The outgassing measurements were performed on a test bench whose schematic view is shown in Figure 2. The system allowed to calculate outgassing rates by throughput method [16]. An orifice of known conductance was interposed between the sample and the inlet of the pumping group and the pressure on both sides of the restriction was monitored using Pirani and Penning gauges. A residual gas analyser (RGA) was mounted above the sample dome to

² The combined surface of the top and bottom faces. Since all the tested samples were very thin, the outgassing from the side walls was considered negligible, therefore only the surface of the two faces has been considered.

conduct a mass spectrometric analysis of the outgassing species. An injection line was connected through a small valve to the sample dome, allowing the venting with nitrogen gas.

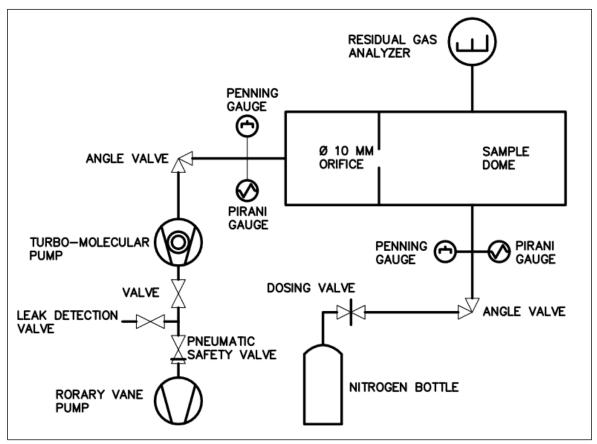


Figure 2: Schematic of the pumping system used for the outgassing measurements

The effective pumping speed of the system, S_{eff} , could be approximated by the conductance of the orifice since the latter was significantly smaller than the pumping speed of the pump [17, p. 93]. In the molecular regime and absence of external leaks, the flow of molecules Q from the sample dome to the inlet of the pumping group can be assumed to be entirely due to the outgassing from the surfaces inside the sample dome, calculated as [18]:

$$Q = C_{tot}(P_{dome} - P_{inlet}) \approx S_{eff} \Delta P \tag{1}$$

A pumping speed of 9.24 L/s was calculated for nitrogen and 11.52 L/s for water vapour. The latter was used in most cases since water was found to be the dominant outgassing species.

3.2 EXPERIMENTAL PROCEDURES

Initially, a pump-down of the empty system was performed (a so-called background measurement), such that the outgassing rates of the walls of the sample dome could be calculated and subtracted from the overall outgassing rates. The samples were tested after being stored in a clean environment, exposed to normal levels of relative humidity (R.H.) present in the air (30-65%) for at least 2 weeks, and except for the cleaning needed for the machined disks, no other operation was performed on them before being introduced into the test bench.

The pump-down procedure was usually carried out at a temperature of about 22° C (295 K), though variations of ca. $\pm 3^{\circ}$ C were observed, due mainly to the day-night cycles or abrupt changes in the external weather conditions. The overall pump-down time was not kept constant among different measurements since the rate at which the pressure decreased inside the dome varied hugely from sample to sample. Each measurement during this phase was therefore interrupted when it was deemed that a sufficiently low pressure had been achieved, or that enough data had been collected, resulting in pump-down times that spanned from 24 hours for the thinnest samples, to over 600 hours for the thickest ones. Near the end of each outgassing measurement, a mass spectrum of the gaseous molecules present in the sample dome was recorded with the RGA.

4 RESULTS

4.1 PUMP-DOWN CURVES AND SPECIFIC OUTGASSING RATES

The pump-down curves measured after the first series of tests, grouped by material type, are shown in Figure 3 in log-log plots. The extrapolated³ pump-down curve of the background, following the $Q \propto t^{-1}$ rule [19, p. 241], is also plotted in each graph as a reference. Initial pressure and outgassing rates were mainly determined by the surface area of the samples, with the Kapton[®] sheets producing the highest outgassing, but within each material they were very similar regardless of the specific thicknesses.

In all cases, the values decreased proportionally to the inverse of the square root of pumping time until a faster decay occurred, visible from a "bend" in the curves which began earlier with the thinnest samples. When the measurements were extended enough time, the pump-down curves of the samples approached asymptotically the curve of the background. This is a sign that their outgassing became eventually negligible compared to the outgassing of the system walls.

Figure 4 displays the plots of the specific outgassing rates of the three materials, again in logarithmic scale, calculated after subtraction of the background data. The outgassing curves of Kapton[®], whose samples were thinner than the other materials, showed very fast decays, occurring in few minutes for the thinnest sheets, and in few hours for the thickest ones. The outgassing rates of PEEK and Vespel[®] exhibited longer decay times relatable to their greater thicknesses

Figure 5 shows the curves obtained from two pairs of PEEK and Vespel® samples with comparable thicknesses. The graph reveals that PEEK exhibits lower initial outgassing rates and earlier decay than Vespel®, given equal surface area and thickness.

The specific outgassing rates of all the samples, measured after 24 hours of pumping, are summarized in Table 2.

³ A sufficiently long empirical pump-down curve for the background was not available at the time of the experiments.

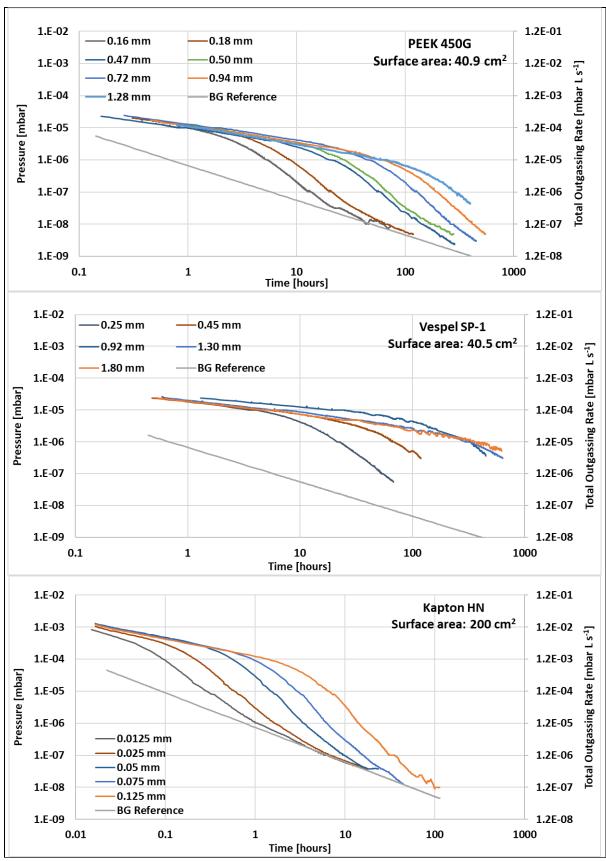


Figure 3: Pump-down curves of the samples collected during the tests

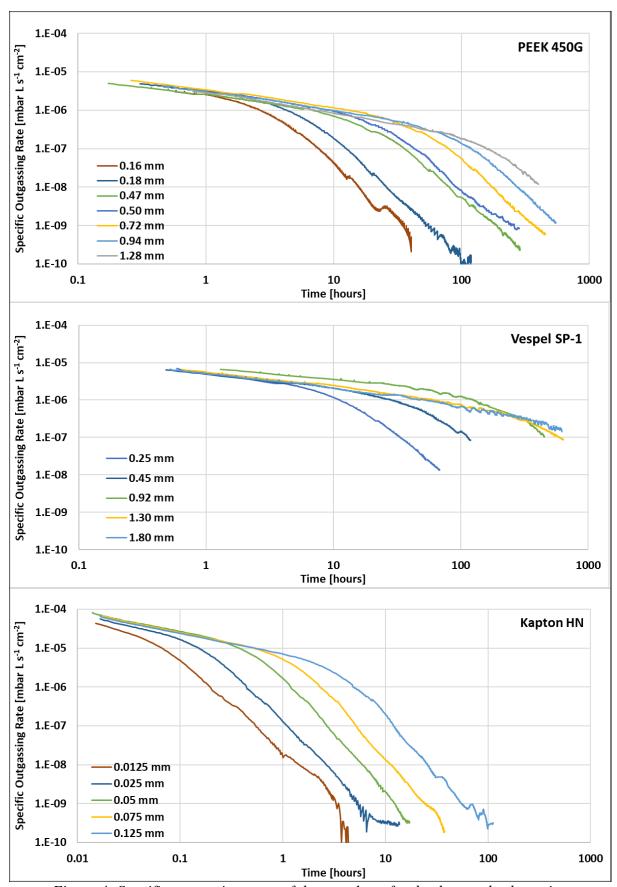


Figure 4: Specific outgassing rates of the samples, after background subtraction

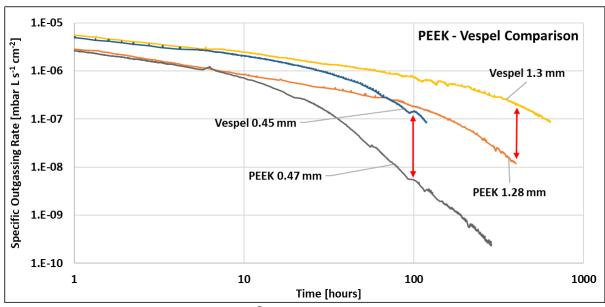


Figure 5: PEEK-Vespel® specific outgassing rates comparison

Table 2: Specific outgassing rates of the tested polymers measured after 24 hours of pumping

PEEK 450G		Vespel® SP-1		Kapton® HN	
Sample	Specific	Sample	Specific	Sample	Specific
thickness	outgassing rate	thickness	outgassing rate	thickness	outgassing rate
[mm]	[mbar L s ⁻¹ cm ⁻²]	[mm]	[mbar L s ⁻¹ cm ⁻²]	[mm]	[mbar L s ⁻¹ cm ⁻²]
0.16	2.4 10-9	0.25	3.1 10 ⁻⁷	0.0125	<10 ⁻¹⁰
0.18	1.4 10-8	0.45	1.5 10 ⁻⁶	0.025	<10 ⁻¹⁰
0.47	2.4 10 ⁻⁷	0.92	2.9 10 ⁻⁶	0.05	6.9 10 ⁻¹⁰
0.5	3.3 10 ⁻⁷	1.3	1.6 10 ⁻⁶	0.075	1.1 10-9
0.72	6.4 10 ⁻⁷	1.8	1.7 10-6	0.125	1.3 10-8
0.94	6.2 10 ⁻⁷				
1.28	5.0 10-7				

4.2 MASS SPECTRA

The mass spectrometry performed at the end of each pump-down confirmed that water vapour was always the dominant outgassing species, as indicated by the peaks at mass 18 amu and 17 amu visible in the spectra of all the samples. Figure 6 shows a selection of these spectra, one for each polymer, normalized with respect to the H_2O peak.

Kapton[®] and PEEK samples produced very similar spectra, with few peaks differing from the ones already visible in the background. For both polymers, the third and fourth strongest signals after H₂O and H₂ (mass 1, 2) were found at mass 28 amu, corresponding to CO and N₂, and at mass 32 amu, corresponding to O₂. The slightly higher peak at 14 amu observed in the PEEK samples, combined with the peak at mass 40 amu (Ar), indicates the presence in this polymer of trapped air.

The spectra of the Vespel® samples showed even higher Ar peaks, as well as the highest peaks at 28, 14 and 7 amu, all due to the high outgassing of air species trapped inside the bulk of the material. This polymer also exhibited a peak at the value 64 amu, corresponding to the outgassing of sulphuric compounds such as S₂ and SO₂, which might be the residual by-

products of the use of aprotic polar solvents such as dimethyl sulfoxide (DMSO) during the synthesis of the polyimide [20]. Across the spectra of most samples, regardless of the polymer type, a small peak at 30 was often found, possibly indicating the release of NO_x molecules.

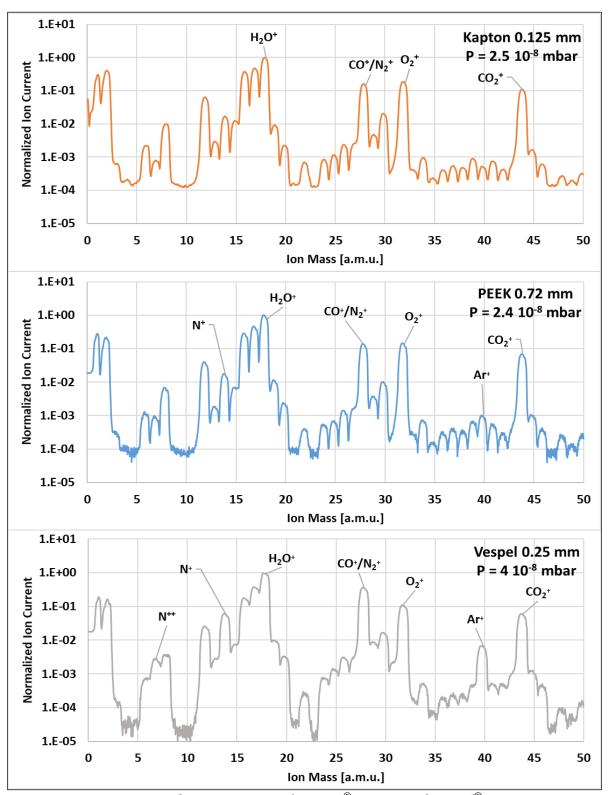


Figure 6: Mass spectra of Kapton®, PEEK and Vespel®

5 DISCUSSION

Since all tested samples had very small thicknesses compared to their other dimensions, they could be considered as infinite thin slabs, for which the flow of water molecules from the bulk to the surface exposed to vacuum can be treated as a mono-dimensional diffusion problem. Assuming that the process is Fickian and that the diffusivity of gas in polymers is constant, the variation of concentration inside the samples during pump-down can be described by the Fick's second law [9, p. 4]:

$$\frac{\partial C}{\partial t} = D \frac{\partial^2 C}{\partial x^2} \tag{2}$$

In the above, C is the concentration of water inside the material, x is the spatial coordinate normal to the faces of the slab, t is pumping time and D is the diffusion coefficient of water in the polymer, which depends on factors such as porosity, crystallinity and chemical structure, and it is different for every material.

With the appropriate boundary conditions describing the exposure to vacuum [19, pp. 247-248], the solution of Equation 2, calculated at the surfaces, is:

$$q = \frac{4D}{h} C_0 \sum_{i=0}^{\infty} exp\left(-\frac{(2i+1)^2 \pi^2 Dt}{h^2}\right)$$
 (3)

where h is the thickness of the slab and C_0 is the initial concentration of water inside the material, assumed uniform at the beginning of the pump-down. Equation 3 can be well approximated by the following two step equation, which are simpler to compute [19, p. 249]:

$$q = \begin{cases} \frac{4D}{h} C_0 \sqrt{\frac{\pi \tau}{16t}} & for \ t \ll 0.5\tau \\ \frac{4D}{h} C_0 e^{-\frac{t}{\tau}} & for \ t \gg 0.5\tau \end{cases}$$
 (4)

where the parameter $\tau = \frac{h^2}{\pi^2 D}$ represents a time constant characterizing the decay of the outgassing rates.

The suitability of the Fickian diffusion model for describing the outgassing of PEEK, Vespel[®] and Kapton[®] was tested by fitting the empirical data with the two-step equation presented above. Plausible values of C_0 (see Section 2) and D [21, p. 16] [22, pp. 156-157] were selected according to the available sources and used as initial guesses for the fitting procedure. A time constant τ based on thickness and diffusion coefficient was then calculated for every sample.

The 2-step equation model worked well for the data collected with most Vespel[®] samples, whose tests, however, were not extended for periods of time much longer than the τ calculated for each specific sample. This was later rectified by increasing the pump-down times when testing most of the PEEK and Kapton[®] samples, in order to collect information on the outgassing behaviour of the polymers for values of $t > 3\tau$. A strong divergence was then observed above this threshold between the empirical data and the model, that regularly

occurred with all samples, when the measurements were extended for long enough times. The 0.25 mm thick Vespel® sample was also retested for a longer time, revealing that the same divergence occurred with all three polymers, as shown in Figure 7.

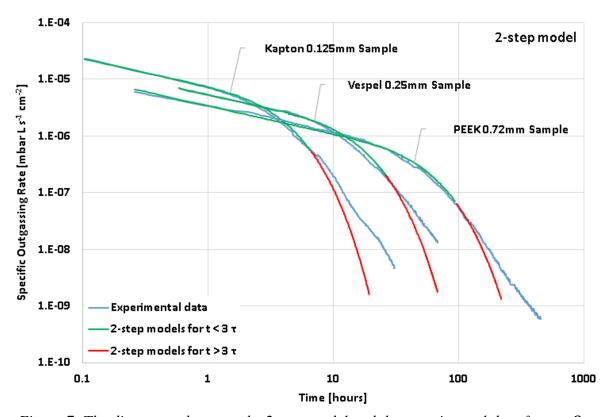


Figure 7: The divergence between the 2-step model and the experimental data for $t > 3\tau$

Regression analysis focused on the final part of the outgassing curves allowed to establish that the relationship $q \propto t^{-3}$ fits very well the data for values of $t > 3\tau$. After writing this relationship in terms of C_0 and τ and rearranging for conveniences some terms in Equation 4, the following 3 equations were assembled and eventually adopted in the study to model the specific outgassing rates of the polymeric samples:

$$q_{face}(t) = \begin{cases} j_0 C_0 \sqrt{\frac{\pi \tau}{t}} & for \ t \le 0.5\tau \\ 4j_0 C_0 e^{-\frac{t}{\tau}} & for \ 0.5\tau < t \le 3\tau \\ \frac{1}{6} j_0 C_0 \left(\frac{\pi \tau}{t}\right)^3 & for \ t > 3\tau \end{cases}$$
 (5)

where the constant $j_0 = \frac{D}{h}$ has been introduced here for ease of calculation.

This 3-step equation eventually allowed to obtain very good fits for all the experimental curves, as it can be seen in Figure 8, showing the specific outgassing rates of the same three samples plotted in Figure 7, this time fitted with the new model.

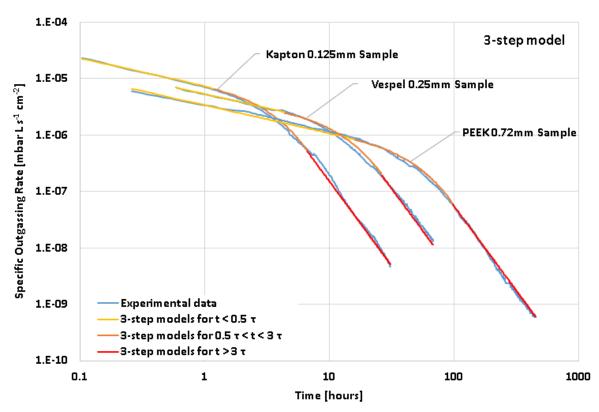


Figure 8: The 3-step model allowed to fit very well the empirical data also for $t > 3\tau$

The optimization of the τ and j_0 parameters carried out during the fitting procedure allowed to calculate the diffusion coefficient D of each sample and compute an average value for each polymer type. Likewise, the initial moisture content C_0 could be estimated for each sample and also in this case an average value was computed for every material. The results of these calculations, which are in good agreement with the data found in the available literature [21] [12] [10] [15] [22], are listed in the Appendix and graphically summarized in Figure 9.

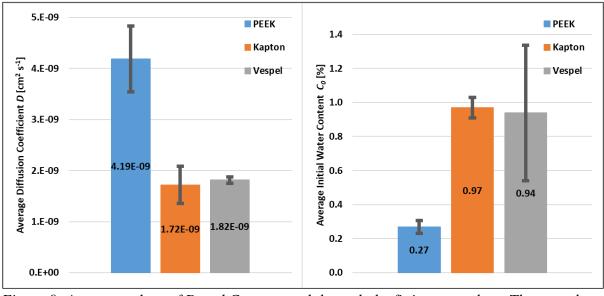


Figure 9: Average values of D and C_0 computed through the fitting procedure. The error bars represent the standard deviation of the data

The PEEK polymer on average exhibited a higher diffusion coefficient and a lower moisture content than Vespel® and Kapton®. This explains its lower outgassing rates and faster decay during pump-down. The average values obtained for the two polyimides were remarkably similar, which might be justified by their common chemical structure (see Section 2). The large standard deviations associated with some of the extrapolated values can be attributed to the small size of the samples available for each material. Furthermore, non-perfectly constant conditions before and during the tests might have contributed to increase the variability of the collected data. Changes in the relative humidity of the air (see Section 3.2) can have influenced the amount of water vapour absorbed by the polymers. Small variations in the environmental temperature (ibid.) could have had a sizable effect on the diffusion coefficients of the samples [19, p. 247].

Despite these uncertainties in the data, the successful application of Equation 4 for modelling the experimental curves allows to draw some general conclusions about the outgassing behaviour of the tested polymers. Higher moisture content causes higher outgassing rates during all stages of the pump-down, while a greater diffusion coefficient, although increasing the initial outgassing rates, makes the latter decay faster, resulting in overall lower outgassing rates, in particular for pumping times beyond 0.5τ . The time constant τ is proportional to the square of the thickness, which is therefore the most important factor affecting the outgassing behaviour of these polymeric materials. Increasing the thickness by only a fraction of a millimetre can potentially extend the necessary pump-down times by hundreds of hours, as it can be seen in Figure 10. Therefore, when considering the use of polymeric components inside HV and UHV systems, the thickness of the components themself should represent the primary concern. Then the values of diffusion coefficient should be as high as possible. Finally, the average moisture content should as low as possible.

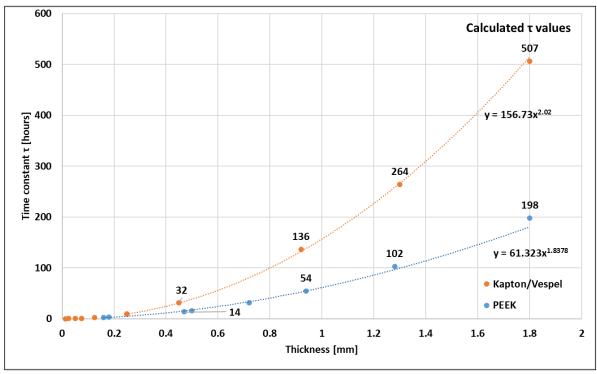


Figure 10: Variation of the time constant τ with the thickness of the tested materials

Regarding the deviation from the standard Fickian diffusion model observed for extended pump-down time, explanations can be sought among the so-called free-volume theories and in particular those involving dual-mode diffusion processes, a general overview of which can be found in Duda and Zielinski [23], while a more detailed study on the subject is provided by Guo et. al. in their work on the kinetics of desorption in glassy polymers [24]. These theories describe a possible dynamic interaction between the molecules dissolved in the semi-crystalline regions of the polymer (Henry population) and the molecules trapped inside micro-voids of the material (Langmuir population). The gas trapped in the micro-voids in the vitreous phase could explain the additional flow of molecules to the surface once low concentrations of the diffusing species are reached. The verification of such hypothesis, however, would require a rigorous mathematical analysis of the model and computer simulations which are beyond the scope of the present work, and will be left for future studies.

As a final note, it is important to remark how the outgassing of atmospheric species such as N₂ and Ar by PEEK and Vespel[®] polymers, revealed by the spectrometric analysis, could be cause of major concerns if detected inside the vacuum chambers of particle accelerators, since it might easily be mistaken for the presence of external air leaks. Vacuum experts and equipment owners must be aware of this possible drawback.

6 CONCLUSIONS

The outgassing rates of several samples of PEEK, Vespel® and Kapton® polymers have been measured through a series of pump-down tests. The experimental data were compared with the outgassing rates predicted by the Fickian diffusion model; agreement between measured and modelled values was found only for relatively short pumping time. A modification of the model was introduced to correctly describe the outgassing rates of these materials for extended pumping time. The analysis allowed to estimate average diffusion coefficients and initial moisture contents of the three polymers, which were in agreement with the values found in the available literature.

Diffusivity, residual content of gas and the thickness of the materials have been correlated with the outgassing behaviour of the polymers during pump-down. The thickness was found to have the major impact on the decay of the outgassing rates, and it should represent the primary concern when selecting polymeric components to be installed into HV or UHV systems, with preference to be given to thinnest available. If choosing between different polymers is possible, those with higher diffusion coefficients and lower expected moisture content should be selected, the first parameter affecting how fast the outgassing rates of the polymers decay, and the latter greatly affecting the magnitude of the outgassing rate during the early stages of pump-down.

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DIFFUSION COEFFICIENTS D, INITIAL MOISTURE CONCENTRATIONS C₀ AND TIME CONSTANTS **\tau** OF THE SAMPLES

APPENDIX

	PEEK				
Material thickness [mm]	D [cm ² s ⁻¹]	C ₀ [%]	τ [h]		
0.16	3.50 10 ⁻⁹	0.250	2.059		
0.18	2.90 10 ⁻⁹	0.330	3.144		
0.47	4.60 10 ⁻⁹	0.220	13.516		
0.5	4.60 10 ⁻⁹	0.280	15.296		
0.72	4.60 10 ⁻⁹	0.300	31.718		
0.94	4.60 10 ⁻⁹	0.280	54.062		
1.28	4.50 10 ⁻⁹	0.220	102.472		
Average	4.19 10 ⁻⁹	0.269			
St. Deviation	6.45 10 ⁻¹⁰	0.038			
	Kapton [®]				
Material thickness [mm]	D [cm ² s ⁻¹]	C ₀ [%]	τ [h]		
0.0125	1.10 10 ⁻⁹	0.900	0.040		
0.025	1.50 10 ⁻⁹	1.000	0.117		
0.05	2.00 10 ⁻⁹	1.050	0.352		
0.075	2.00 10 ⁻⁹	1.000	0.792		
0.125	2.00 10 ⁻⁹	0.900	2.199		
Average	1.72 10 ⁻⁹	0.970			
St. Deviation	3.66 10 ⁻¹⁰	0.060			
	Vespel [®]				
Material thickness [mm]	D [cm ² s ⁻¹]	C ₀ [%]	τ [h]		
0.25	1.80 10 ⁻⁹	0.680	9.772		
0.25	1.95 10 ⁻⁹	0.650	9.021		
0.45	1.80 10 ⁻⁹	0.750	31.663		
0.92	1.75 10 ⁻⁹	1.800	136.124		
1.3	1.80 10 ⁻⁹	0.950	264.248		
1.8	1.80 10 ⁻⁹	0.800	506.606		
Average	1.82 10 ⁻⁹	0.938			
St. Deviation	5.77 10 ⁻¹¹	0.368			